Pollutant	Reference or	Manual or auto-	Applicable part 50 appen-	Applicable subparts of part 53					
Pollularii	equivalent	mated	dix	Α	В	С	D	E	F
	Equivalent	Manual	Q	1		1			
		Automated	Q	1		1			
PM ₁₀	Reference	Manual	J	1			1		
	Equivalent	Manual	J	1		/	1		
		Automated	J	1		1	1		
PM _{2.5}	Reference	Manual	L	1				/	
	Equivalent Class I	Manual	L	1		1		1	
	Equivalent Class II	Manual	L¹	1		✓2		1	/ 12
	Equivalent Class	Automated	ני	1		1		1	✓¹
PM _{10-2.5}	Reference	Manual	L, O	1				/	
	Equivalent Class I	Manual	L, O	1		/		/	
	Equivalent Class II	Manual	L, O	1		✓2		1	√ 12
	Equivalent Class	Automated	L1, O1	1		1		1	✓1
	III.								

^{1.} Some requirements may apply, based on the nature of each particular candidate method, as determined by the Administrator.

[75 FR 35597, June 22, 2010]

APPENDIX A TO SUBPART A OF PART 53— REFERENCES

- (1) American National Standard Quality Systems—Model for Quality Assurance in Design, Development, Production, Installation, and Servicing, ANSI/ISO/ASQC Q9001—1994. Available from American Society for Quality, P.O. Box 3005, Milwaukee, WI 53202 (http://qualitypress.asg.org).
- (2) American National Standard Quality Systems for Environmental Data and Technology Programs—Requirements with guidance for use, ANSI/ASQC E4-2004. Available from American Society for Quality P.O. Box 3005, Milwaukee, WI 53202 (http://qualitypress.asq.org).
- (3) Dimensioning and Tolerancing, ASME Y14.5M-1994. Available from the American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017.
- (4) Mathematical Definition of Dimensioning and Tolerancing Principles, ASME Y14.5.1M-1994. Available from the American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017.
- (5) ISO 10012, Quality Assurance Requirements for Measuring Equipment-Part 1: Meteorological confirmation system for measuring equipment):1992(E). Available from American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee, WI 53202.
- (6) Quality Assurance Guidance Document 2.12. Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. EPA, National Exposure Research Laboratory, Research Triangle Park, NC, November 1998 or later edition. Currently available at http://www.epa.gov/ttn/amtic/pmqainf.html.

[62 FR 38784, July 18, 1997, as amended at 71 FR 61278, Oct. 17, 2006]

Subpart B—Procedures for Testing Performance Characteristics of Automated Methods SO₂, CO, O₃, and NO₂

§53.20 General provisions.

- (a) The test procedures given in this subpart shall be used to test the performance of candidate automated methods against the performance specifications given in table B-1. A test analyzer representative of the candidate automated method must exhibit performance better than, or equal to, the specified value for each such specification (except Range) to satisfy the requirements of this subpart. Except as provided in paragraph (b) of this section, the range of the candidate method must be the range specified in table B-1 to satisfy the requirements of this subpart.
- (b) For a candidate method having more than one selectable measurement range, one range must be that specified in table B-1 (standard range for SO₂), and a test analyzer representative of the method must pass the tests required by this subpart while operated in that range. The tests may be repeated for one or more broader ranges (i.e., ones extending to higher concentrations) than the range specified in table B-1, provided that the range does not extend to concentrations more than four times the upper range limit specified in table B-1. For broader

Alternative Class III requirements may be substituted.

ranges, only the tests for range (calibration), noise at 80% of the upper range limit, and lag, rise and fall time are required to be repeated. The tests may be repeated for one or more narrower ranges (ones extending to lower concentrations) than that specified in table B-1. For SO₂ methods, table B-1 specifies special performance requirements for narrower (lower) ranges. For methods other than SO₂, only the tests for range (calibration), noise at 0% of the measurement range, and lower detectable limit are required to be repeated. If the tests are conducted or passed only for the specified range (standard range for SO₂), any FRM or FEM method determination with respect to the method will be limited to that range. If the tests are passed for both the specified range and one or more broader ranges, any such determination will include the additional range(s) as well as the specified range, provided that the tests required by subpart C of this part (if applicable) are met for the broader range(s). If the tests are passed for both the specified range and one or more narrower ranges, any FRM or FEM method determination for the method will include the narrower range(s) as well as

the specified range. Appropriate test data shall be submitted for each range sought to be included in a FRM or FEM method determination under this paragraph (b).

- (c) For each performance specification (except Range), the test procedure shall be initially repeated seven (7) times to yield 7 test results. Each result shall be compared with the corresponding specification in table B-1; a value higher than or outside that specified constitutes a failure. These 7 results for each parameter shall be interpreted as follows:
- (1) Zero (0) failures: Candidate method passes the performance parameter.
- (2) Three (3) or more failures: Candidate method fails the performance parameter.
- (3) One (1) or two (2) failures: Repeat the test procedures for the parameter eight (8) additional times yielding a total of fifteen (15) test results. The combined total of 15 test results shall then be interpreted as follows:
- (i) One (1) or two (2) failures: Candidate method passes the performance parameter.
- (ii) Three (3) or more failures: Candidate method fails the performance parameter.

TABLE B-1—PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS

		SC)2				Definitions and
Performance parameter	Units 1	Std. range ³	Lower range 23	O ₃	СО	NO ₂	test procedures
Range Noise Lower detectable limit Interference equivalent	ppm ppm	0–0.5 0.001 0.002	<0.5 0.0005 0.001	0–0.5 0.005 0.010	0–50 0.5 1.0	0-0.5 0.005 0.010	Sec. 53.23(a). Sec. 53.23(b). Sec. 53.23(c).
Each interferent	ppm	±0.005	4±0.005	±0.02	±1.0	±0.02	Sec. 53.23(d).
Total, all interferents	ppm	_	_	0.06	1.5	0.04	Sec. 53.23(d).
5. Zero drift, 12 and 24 hour	ppm	±0.004	±0.002	±0.02	±1.0	±0.02	Sec. 53.23(e).
Span drift, 24 hour							
20% of upper range limit	Percent	_	_	±20.0	±10.0	±20.0	Sec. 53.23(e).
80% of upper range limit	Percent	±3.0	±3.0	±5.0	±2.5	±5.0	Sec. 53.23(e).
7. Lag time	Minutes	2	2	20	10	20	Sec. 53.23(e).
8. Rise time	Minutes	2	2	15	5	15	Sec. 53.23(e).
9. Fall time	Minutes	2	2	15	5	15	Sec. 53.23(e).
20% of upper range limit	ppm	_	_	0.010	0.5	0.020	Sec. 53.23(e).
spportange iiiii	Percent	2	2			0.020	Sec. 53.23(e).
80% of upper range limit	ppm			0.010	0.5	0.030	Sec. 53.23(e).
	Percent	2	2				Sec. 53.23(e).

^{1.} To convert from parts per million (ppm) to $\mu g/m^3$ at 25 °C and 760 mm Hg, multiply by M/0.02447, where M is the molecular

 ^{1.} To convert from parts per million (ppm) to μg/m² at 25 °C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas. Percent means percent of the upper range limit.
 2. Tests for interference equivalent and lag time do not need to be repeated for any lower SO₂ range provided the test for the standard range shows that the lower range specification is met for each of these test parameters.
 3. For candidate analyzers having automatic or adaptive time constants or smoothing filters, describe their functional nature, and describe and conduct suitable lests to demonstrate their function aspects and verify that performances for calibration, noise, lag, rise, fall times, and precision are within specifications under all applicable conditions. For candidate analyzers with operator-selectable time constants or smoothing filters, conduct calibration, noise, lag, rise, fall times, and precision tests at the highest and lowest settings that are to be included in the FRM or FEM designation.
 4. For nitric oxide interference for the SO₂ UVF method, interference equivalent is ±0.003 ppm for the lower range.

- (d) The tests for zero drift, span drift, lag time, rise time, fall time, and precision shall be combined into a single sequential procedure to be conducted at various line voltages and ambient temperatures specified in §53.23(e). The tests for noise, lower detectable limit, and interference equivalents shall be made at any temperature between 20 °C. and 30 °C. and at any normal line voltage between 105 and 125 volts, and shall be conducted such that not more than three (3) test results for each parameter are obtained per 24 hours.
- (e) All response readings to be recorded shall first be converted to concentration units according to the calibration curve constructed in accordance with §53.21(b).
- (f) All recorder chart tracings, records, test data and other documentation obtained from or pertinent to these tests shall be identified, dated, signed by the analyst performing the test, and submitted.

Note: Suggested formats for reporting the test results and calculations are provided in Figures B-2, B-3, B-4, B-5, and B-6 in appendix A. Symbols and abbreviations used in this subpart are listed in table B-5, appendix A.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 41 FR 52694, Dec. 1, 1976; 75 FR 35598, June 22, 2010]

§ 53.21 Test conditions.

(a) Set-up and start-up of the test analyzer shall be in strict accordance with the operating instructions specified in the manual referred to in §53.4(b)(3). Allow adequate warm-up or stabilization time as indicated in the operating instructions before beginning the tests. The test procedures assume that the test analyzer has an analog measurement signal output that is connected to a suitable strip chart recorder of the servo, null-balance type. This recorder shall have a chart width of a least 25 centimeters, chart speeds up to 10 cm per hour, a response time of 1 second or less, a deadband of not more than 0.25 percent of full scale, and capability either of reading measurements at least 5 percent below zero or of offsetting the zero by at least 5 percent. If the test analyzer does not have an analog signal output, or if other types of measurement data output are used, an alternative measurement data recording device (or devices) may be used for the tests, provided it is reasonably suited to the nature and purposes of the tests and an analog representation of the analyzer measurements for each test can be plotted or otherwise generated that is reasonably similar to the analog measurement recordings that would be produced by a conventional chart recorder.

- (b) Calibration of the test analyzer shall be as indicated in the manual referred to in §53.4(b)(3) and as follows: If the chart recorder does not have below zero capability, adjust either the controls of the test analyzer or the chart recorder to obtain a +5% offset zero reading on the recorder chart to facilitate observing negative response or drift. If the candidate method is not capable of negative response, the test analvzer (not recorder) shall be operated with an offset zero. Construct and submit a calibration curve showing a plot of recorder scale readings (ordinate) against pollutant concentrations (abscissa). A plot of output units (volts, millivolts, milliamps, etc.) against pollutant concentrations shall also be shown for methods not including an integral chart recorder. All such plots shall consist of at least seven (7) approximately equally spaced, identifiable points, including 0 and 90±5 percent of full scale.
- (c) Once the test analyzer has been set up and calibrated and the tests started, manual adjustment or normal periodic maintenance is permitted only every 3 days. Automatic adjustments which the test analyzer performs by itself are permitted at any time. The submitted records shall show clearly when any manual adjustment or periodic maintenance was made and describe the operations performed.
- (d) If the test analyzer should malfunction during any of the performance tests, the tests for that parameter shall be repeated. A detailed explanation of the malfunction, remedial action taken, and whether recalibration was necessary (along with all pertinent records and charts) shall be submitted. If more than one malfunction occurs, all performance test procedures for all parameters shall be repeated.

(e) Tests for all performance parameters shall be completed on the same test analyzer, except that use of multiple test analyzers to accelerate testing will be permitted when alternate ranges of a multi-range candidate method are being tested.

[62 FR 38784, July 18, 1997, as amended at 75 FR 35599, June 22, 2010]

§ 53.22 Generation of test atmospheres.

(a) Table B-2 specifies preferred methods for generating test atmospheres and suggested methods of verifying the concentrations. Only one means of establishing the concentration of a test atmosphere is normally required. If the method of generation can produce reproducible concentrations, verification is optional. If the method of generation is not reproducible, then establishment of the concentration by some verification meth-

od is required. However, when a method of generation other than that given in table B-2 is used, the test concentration shall be verified.

- (b) The test atmosphere delivery system shall be designed and constructed so as not to significantly alter the test atmosphere composition or concentration during the period of the test. The delivery system shall be fabricated from borosilicate glass or FEP Teflon.
- (c) The output of the test atmosphere generation system shall be sufficiently stable to obtain stable response during the required tests. If a permeation device is used for generation of a test atmosphere, the device, as well as the air passing over it, shall be controlled to $\pm 0.1~^{\circ}\mathrm{C}$.
- (d) All diluent air shall be zero air free of contaminants likely to cause a detectable response on the test analyzer.

TABLE B-2-TEST ATMOSPHERES

Test gas	Generation	Verification
Ammonia	Permeation device. Similar to system described in references 1 and 2.	Indophenol method, reference 3.
Carbon dioxide	Cylinder of zero air or nitrogen containing CO_2 as required to obtain the concentration specified in Table B–3.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.
Carbon monoxide	Cylinder of zero air or nitrogen containing CO as required to obtain the concentration specified in Table B–3.	Use a FRM CO analyzer as described in reference 8.
Ethane	Cylinder of zero air or nitrogen containing eth- ane as required to obtain the concentration specified in Table B–3.	Gas chromatography, ASTM D2820, reference 10. Use NIST-traceable gaseous methane or propane standards for calibration.
Ethylene	Cylinder of pre-purified nitrogen containing ethylene as required to obtain the concentration specified in Table B-3.	Do.
Hydrogen chloride	Cylinder¹ of pre-purified nitrogen containing approximately 100 ppm of gaseous HCL. Di- lute with zero air to concentration specified in Table B-3.	Collect samples in bubbler containing distilled water and analyze by the mercuric thiocyante method, ASTM (D612), p. 29, reference 4.
Hydrogen sulfide	Permeation device system described in references 1 and 2.	Tentative method of analysis for H ₂ S content of the atmosphere, p. 426, reference 5.
Methane	Cylinder of zero air containing methane as re- quired to obtain the concentration specified in Table B–3.	Gas chromatography ASTM D2820, reference 10. Use NIST-traceable methane standards for calibration.
Naphthalene	Permeation device as described in references 1 and 2. Cylinder of pre-purified nitrogen containing 100 ppm naphthalene. Dilute with zero air to concentration specified in Table B–3.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.

TABLE B-2-TEST ATMOSPHERES-Continued

Test gas	Generation	Verification
Nitric oxide	Cylinder¹ of pre-purified nitrogen containing approximately 100 ppm NO. Dilute with zero air to required concentration.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.
Nitrogen dioxide	Gas phase titration as described in reference 6. Permeation device, similar to system described in reference 6.	Use an FRM NO ₂ analyzer calibrated with a gravimetrically calibrated permeation device. Use an FRM NO ₂ analyzer calibrated by gas-phase titration as described in reference 6.
Ozone	Calibrated ozone generator as described in reference 9.	Use an FEM ozone analyzer calibrated as described in reference 9.
Sulfur dioxide	Permeation device as described in ref- erences 1 and 2. Dynamic dilution of a cylinder containing ap- proximately 100 ppm SO ₂ as described in	Use an SO ₂ FRM or FEM analyzer as described in reference 7.
Water	Reference 7. Pass zero air through distilled water at a fixed known temperature between 20° and 30 °C such that the air stream becomes saturated. Dilute with zero air to concentration specified in Table B-3.	Measure relative humidity by means of a dew- point indicator, calibrated electrolytic or piezo electric hygrometer, or wet/dry bulb thermometer.
Xylene	Cylinder of pre-purified nitrogen containing 100 ppm xylene. Dilute with zero air to concentration specified in Table B–3.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.
Zero air	Ambient air purified by appropriate scrubbers or other devices such that it is free of contaminants likely to cause a detectable response on the analyzer. Cylinder of compressed zero air certified by the supplier or an independent laboratory to be free of contaminants likely to cause a detectable response on the analyzer.	

- (e) The concentration of each test atmosphere shall be established and/or verified before or during each series of tests. Samples for verifying test concentrations shall be collected from the test atmosphere delivery system as close as possible to the sample intake port of the test analyzer.
- (f) The accuracy of all flow measurements used to calculate test atmosphere concentrations shall be documented and referenced to a primary standard (such as a spirometer, bubble meter, etc.). Any corrections shall be clearly shown. All flow measurements

¹ Use stainless steel pressure regulator dedicated to the pollutant measured.
Reference 1. O'Keefe, A. E., and Ortaman, G. C. "Primary Standards for Trace Gas Analysis," *Anal. Chem.* 38, 760 (1966).
Reference 2. Scaringelli, F. P., A. E. Rosenberg, E., and Bell, J. P., "Primary Standards for Trace Gas Analysis." *Anal. Chem.* 38, 760 (1966).

Reference 3. "Tentative Method of Analysis for Ammonia in the Atmosphere (Indophenol Method)", Health Lab Sciences, vol. 10, No. 2, 115–118, April 1973.

Reference 4. 1973 Annual Book of ASTM Standards, American Society for Testing and Materials, 1916 Race St., Philadelphia,

PA.
Reference 5. Methods for Air Sampling and Analysis, Intersociety Committee, 1972, American Public Health Association, 1015.
Reference 6. 40 CFR 50 Appendix F, "Measurement Principle and Calibration Principle for the Measurement of Nitrogen Dioxide in the Atmosphere (Gas Phase Chemiluminescence)."
Reference 7. 40 CFR 50 Appendix A-1, "Measurement Principle and Calibration Procedure for the Measurement of Sulfur Dioxide in the Atmosphere (Ultraviolet Fluorscence)."
Reference 8. 40 CFR 50 Appendix C, "Measurement Principle and Calibration Procedure for the Measurement of Carbon Monoxide in the Atmosphere" (Non-Dispersive Infrared Photometry)".
Reference 9. 40 CFR 50 Appendix D, "Measurement Principle and Calibration Procedure for the Measurement of Ozone in the Atmosphere" (Non-Dispersive Infrared Photometry)".
Reference 10. "Standard Test Method for C, through C5 Hydrocarbons in the Atmosphere by Gas Chromatography", D 2820,

Reference 10. "Standard Test Method for C, through C5 Hydrocarbons in the Atmosphere by Gas Chromatography", D 2820, 1987 Annual Book of Aston Standards, vol 11.03, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA

§ 53.23

given in volume units shall be standardized to $25~^{\circ}$ C. and 760~mm Hg.

(g) Schematic drawings and other information showing complete procedural details of the test atmosphere generation, verification, and delivery system shall be provided. All pertinent calculations shall be clearly indicated.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 75 FR 35599, June 22, 2010]

§ 53.23 Test procedures.

(a) Range—(1) Technical definition. Nominal minimum and maximum concentrations which a method is capable of measuring.

NOTE: The nominal range is specified at the lower and upper range limits in concentration units, for example, $0-0.5~\rm p/m$.

(2) Test procedure. Submit a suitable calibration curve, as specified in §53.21(b), showing the test analyzer's response over at least 95 percent of the required range.

Note: A single calibration curve will normally suffice.

- (b) Noise—(1) Technical definition. Spontaneous, short duration deviations in output, about the mean output, which are not caused by input concentration changes. Noise is determined as the standard deviation about the mean and is expressed in concentration units.
- (2) Test procedure. (i) Allow sufficient time for the test analyzer to warm up and stabilize. Determine at two concentrations, first using zero air and then a pollutant test gas concentration as indicated below. The noise specification in table B-1 shall apply to both of these tests.
- (ii) Connect an integrating-type digital meter (DM) suitable for the test analyzer's output and accurate to three significant digits, to measure the analyzer's output signal.

NOTE: Use of a chart recorder in addition to the DM is optional.

- (iii) Measure zero air for 60 minutes. During this 60-minute interval, record twenty-five (25) readings at 2-minute intervals. (See Figure B-2 in appendix A.)
- (iv) Convert each DM reading to concentration units (p/m) by reference to

the test analyzer's calibration curve as determined in §53.21(b). Label the converted DM readings $r_1, r_2, r_3 \ldots r_i \ldots r_{25}$.

(v) Calculate the standard deviation, S, as follows:

$$S = \sqrt{\frac{\sum_{i=1}^{25} (r_i)^2 - \frac{1}{25} \left(\sum_{i=1}^{25} r_i\right)^2}{24}} (p / m)$$

where i indicates the i-th DM reading in ppm.

- (vi) Let S at 0 ppm be identified as S_o ; compare S_o to the noise specification given in table B-1.
- (vii) Repeat steps (iii) through (vi) of this section using a pollutant test atmosphere concentration of 80 ± 5 percent of the upper range limit (URL) instead of zero gas, and let S at 80 percent of the URL be identified as S_{80} . Compare S_{80} to the noise specification given in table B–1.
- (viii) Both S_0 and S_{80} must be less than or equal to the specification for noise to pass the test for the noise parameter.
- (c) Lower detectable limit—(1) Technical definition. The minimum pollutant concentration which produces a signal of twice the noise level.
- (2) Test procedure. (i) Allow sufficient time for the test analyzer to warm up and stabilize. Measure zero air and record the stable reading in ppm as B_Z . (See Figure B–3 in appendix A.)
- (ii) Generate and measure a pollutant test atmosphere concentration equal to the value for the lower detectable limit specified in table B-1.

NOTE: If necessary, the test atmosphere concentration may be generated or verified at a higher concentration, then accurately diluted with zero air to the final required concentration.

- (iii) Record the test analyzer's stable indicated reading, in ppm, as $B_{\rm L}$.
- (iv) Determine the Lower Detectable Limit (LDL) as LDL = $B_{\rm L}-B_{\rm Z}$. Compare this LDL value with the noise level, S_0 , determined in §53.23(b), for 0 concentration test atmosphere. LDL must be equal to or higher than $2S_0$ to pass this test.

- (d) Interference equivalent—(1) Technical definition. Positive or negative response caused by a substance other than the one being measured.
- (2) Test procedure. The test analyzer shall be tested for all substances likely to cause a detectable response. The test analyzer shall be challenged, in turn, with each interfering agent specified in table B-3. In the event that there are substances likely to cause a significant interference which have not been specified in table B-3, these substances shall be tested at a concentration substantially higher than that normally found in the ambient air. The interference may be either positive or negative, depending on whether the test analyzer's response is increased or decreased by the presence of the interferent. Interference equivalents

shall be determined by mixing each interferent, one at a time, with the pollutant at the concentrations specified in table B-3, and comparing the test analyzer's response to the response caused by the pollutant alone. Known gas-phase reactions that might occur between an interferent and the pollutant are designated by footnote 3 in table B-3. In these cases, the interference equivalent shall be determined in the absence of the pollutant.

- (i) Allow sufficient time for warm-up and stabilization of the test analyzer.
- (ii) For a candidate method using a prefilter or scrubber based upon a chemical reaction to derive part of its specificity, and which requires periodic service or maintenance, the test analyzer shall be "conditioned" prior to each interference test as follows:

TABLE B-3—INTERFERENT TEST CONCENTRATION, 1 PARTS PER MILLION

Maybrace type Hydro-ploted acide Sulfure acide Sulfure acide Number of ploted acide Sulfure acide Number of ploted acide Inflict Carbon acide Hydro-socration acide Purple acide Number occurred acide Market acide																	
50.1 40.14 0.5 0.5 750 0.5 20,000 50 0.2 0.01 40.14 0.5 750 0.5 20,000 50 0.2 0.1 40.14 0.5 <td< th=""><th></th><th>Analyzer type</th><th>Hydro- chloric acid</th><th>Ammo- nia</th><th>Hydro- gen sulfide</th><th>Sulfur dioxide</th><th>Nitro- gen dioxide</th><th>Nitric oxide</th><th>Carbon dioxide</th><th>Ethy- lene</th><th>Ozone</th><th>M- xy- lene</th><th>Water</th><th>Carbon mon- oxide</th><th>Meth- ane</th><th>Ethane</th><th>Naph- thalene</th></td<>		Analyzer type	Hydro- chloric acid	Ammo- nia	Hydro- gen sulfide	Sulfur dioxide	Nitro- gen dioxide	Nitric oxide	Carbon dioxide	Ethy- lene	Ozone	M- xy- lene	Water	Carbon mon- oxide	Meth- ane	Ethane	Naph- thalene
0.02 40.14 750 750 320,000 50 0.2 0.1 40.14 0.5 0.5 750 0.5 320,000 50 0.2 0.1 0.1 40.14 0.5 0.5 750 0.5 <t< td=""><td>Ultra</td><td>escence</td><td></td><td></td><td>50.1</td><td>40.14</td><td>0.5</td><td>0.5</td><td></td><td></td><td>0.5</td><td>0.2</td><td>20,000</td><td></td><td></td><td></td><td>60.05</td></t<>	Ultra	escence			50.1	40.14	0.5	0.5			0.5	0.2	20,000				60.05
0.2 0.1 40.14 0.5 0.5 750 0.5 </td <td>Flai</td> <td>me photometric</td> <td>:</td> <td></td> <td>0.01</td> <td>40.14</td> <td></td> <td></td> <td>750</td> <td></td> <td></td> <td></td> <td>320,000</td> <td>20</td> <td></td> <td></td> <td></td>	Flai	me photometric	:		0.01	40.14			750				320,000	20			
0.2 0.1 0.1 40.14 0.5 0.5 0.2 0.5 0.5 0.5 0.0 0.5 0.0 0.5 0.0 </td <td>ga</td> <td>s chromatography</td> <td></td> <td></td> <td>0.1</td> <td>40.14</td> <td></td> <td>:</td> <td>750</td> <td>:</td> <td></td> <td></td> <td>320,000</td> <td>20</td> <td></td> <td></td> <td></td>	ga	s chromatography			0.1	40.14		:	750	:			320,000	20			
0.2 0.1 40.14 0.5 0.5 750 0.2 0.5 </td <td>Spe</td> <td>ectrophotometric-wet hemical (pararosanaline).</td> <td>0.2</td> <td>0.1</td> <td>0.1</td> <td>40.14</td> <td>0.5</td> <td></td> <td>750</td> <td></td> <td>0.5</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	Spe	ectrophotometric-wet hemical (pararosanaline).	0.2	0.1	0.1	40.14	0.5		750		0.5						
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	Š	pectrophotometric-gas		30.1		0.5	40.1	0.5			0.5		20,000	20			

1. Concentrations of interferent listed must be prepared and controlled to ±10 percent of the stated value.

2. Analyzer types not listed will be considered by the Administrator as special cases.

3. Do not mix with the pollutant.

4. Concentration of pollutant used for test. These pollutant concentrations must be prepared to ±10 percent of the stated value.

5. If candidate method utilizes an elevated-temperature scrubber for removal of aromatic hydrocarbons, perform this interference test.

6. If candidate method utilizes an elevated-temperature scrubber for removal of aromatic hydrocarbons, use a test concentration cannot be accurately quantified, remove the scrubber, use a test concentration that causes a full scale response, reattach the scrubber, and evaluate response for interference

- (A) Service or perform the indicated maintenance on the scrubber or prefilter as directed in the manual referred to in §53.4(b)(3).
- (B) Before testing for each interferent, allow the test analyzer to sample through the scrubber a test atmosphere containing the interferent at a concentration equal to the value specified in table B-3. Sampling shall be at the normal flow rate and shall be continued for 6 continuous hours prior to testing.
- (iii) Generate three test atmosphere streams as follows:
- (A) Test atmosphere *P*: Pollutant concentration.
- (B) Test atmosphere *I*: Interference concentration.
 - (C) Test atmosphere Z: Zero air.
- (iv) Adjust the individual flow rates and the pollutant or interferent generators for the three test atmospheres as follows:
- (A) The flow rates of test atmospheres I and Z shall be identical.
- (B) The concentration of pollutant in test atmosphere P shall be adjusted such that when P is mixed (diluted) with either test atmosphere I or Z, the resulting concentration of pollutant shall be as specified in table B-3.
- (C) The concentration of interferent in test atmosphere I shall be adjusted such that when I is mixed (diluted) with test atmosphere P, the resulting concentration of interferent shall be equal to the value specified in table B-3.
- (D) To minimize concentration errors due to flow rate differences between I and Z, it is recommended that, when possible, the flow rate of P be from 10 to 20 times larger than the flow rates of I and Z.
- (v) Mix test atmospheres P and Z by passing the total flow of both atmospheres through a mixing flask.
- (vi) Sample and measure the mixture of test atmospheres P and Z with the test analyzer. Allow for a stable reading, and record the reading, in concentration units, as R (see Figure B-3).
- (vii) Mix test atmospheres P and I by passing the total flow of both atmospheres through a mixing flask.
- (viii) Sample and measure this mixture. Record the stable reading, in concentration units, as R_I .

(ix) Calculate the interference equivalent (IE) as:

 $IE = R_I - R$

IE must be equal to or less than the specification given in table B-1 for each interferent to pass the test.

- (x) Follow steps (iii) through (ix) of this section, in turn, to determine the interference equivalent for each interferent.
- (xi) For those interferents which cannot be mixed with the pollutant, as indicated by footnote (3) in table B-3, adjust the concentration of test atmosphere I to the specified value without being mixed or diluted by the pollutant test atmosphere. Determine IE as follows:
- (A) Sample and measure test atmosphere Z (zero air). Allow for a stable reading and record the reading, in concentration units, as R.
- (B) Sample and measure the interferent test atmosphere I. If the test analyzer is not capable of negative readings, adjust the analyzer (not the recorder) to give an offset zero. Record the stable reading in concentration units as R_I , extrapolating the calibration curve, if necessary, to represent negative readings.
- (C) Calculate $IE=R_I-R$. IE must be equal to or less than the specification in table B-1 to pass the test.
- (xii) Sum the absolute value of all the individual interference equivalents. This sum must be equal to or less than the total interferent specification given in table B-1 to pass the test.
- (e) Zero drift, span drift, lag time, rise time, fall time, and precision—(1) Technical definitions—(i) Zero drift: The change in response to zero pollutant concentration, over 12- and 24-hour periods of continuous unadjusted operation.
- (ii) Span drift: The percent change in response to an up-scale pollutant concentration over a 24-hour period of continuous unadjusted operation.
- (iii) Lag time: The time interval between a step change in input concentration and the first observable corresponding change in response.
- (iv) *Rise time*: The time interval between initial response and 95 percent of final response after a step increase in input concentration.

- (v) Fall time: The time interval between initial response and 95 percent of final response after a step decrease in input concentration.
- (vi) *Precision:* Variation about the mean of repeated measurements of the same pollutant concentration, expressed as one standard deviation about the mean.
- (2) Tests for these performance parameters shall be accomplished over a period of seven (7) or more days. During this time, the line voltage supplied to the test analyzer and the ambient temperature surrounding the analyzer shall be varied from day to day. One test result for each performance parameter shall be obtained each test day, for seven (7) or fifteen (15) test days as necessary. The tests are performed sequentially in a single procedure.
- (3) The 24-hour test day may begin at any clock hour. The first 12 hours out of each test day are required for testing 12-hour zero drift. Tests for the other parameters shall be conducted during the remaining 12 hours.
- (4) Table B-4 specifies the line voltage and room temperature to be used for each test day. The line voltage and temperature shall be changed to the specified values at the start of each test day (i.e., at the start of the 12-hour zero test). Initial adjustments (day zero) shall be made at a line voltage of 115 volts (rms) and a room temperature of 25 °C.
- (5) The tests shall be conducted in blocks consisting of 3 test days each until 7 or 15 test results have been obtained. (The final block may contain fewer than three test days.) If a test is interrupted by an occurrence other

than a malfunction of the test analyzer, only the block during which the interruption occurred shall be repeated.

- (6) During each block, manual adjustments to the electronics, gas, or reagent flows or periodic maintenance shall not be permitted. Automatic adjustments which the test analyzer performs by itself are permitted at any time.
- (7) At least 4 hours prior to the start of the first test day of each block, the test analyzer may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual referred to in §53.4(b)(3). If a new block is to immediately follow a previous block, such adjustments or servicing may be done immediately after completion of the day's tests for the last day of the previous block and at the voltage and temperature specified for that day, but only on test days 3, 6, 9, and 12.

NOTE: If necessary, the beginning of the test days succeeding such maintenance or adjustment may be delayed as necessary to complete the service or adjustment operation.

(8) All response readings to be recorded shall first be converted to concentration units according to the calibration curve. Whenever a test atmosphere is to be measured but a stable reading is not required, the test atmosphere shall be measured long enough to cause a change in response of at least 10% of full scale. Identify all readings and other pertinent data on the strip chart. (See Figure B-1 illustrating the pattern of the required readings.)

TABLE B-4—LINE VOLTAGE AND ROOM	TEMPERATURE TEST CONDITIONS
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Test day	Line volt- age, 1 rms	Room tem- perature, ² °C	Comments
0	115	25	Initial set-up and adjustments.
1	125	20	
2	105	20	
3	125	30	Adjustments and/or periodic maintenance permitted at end of tests.
4	105	30	
5	125	20	
6	105	20	Adjustments and/or periodic maintenance permitted at end of tests.
7	125	30	Examine test results to ascertain if further testing is required.
8	105	30	
9	125	20	Adjustments and/or periodic maintenance permitted at end of tests.
10	105	20	
11	125	30	
12	105	30	Adjustments and/or periodic maintenance permitted at end of tests.

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TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS—Continued

Test day	Line volt- age, 1 rms	Room tem- perature, ² °C	Comments
13	125	20	
14	105	20	
15	125	30	

 $^{^1}$ Voltage specified shall be controlled to ± 1 volt. 2 Temperature specified shall be controlled to ± 1 °C.

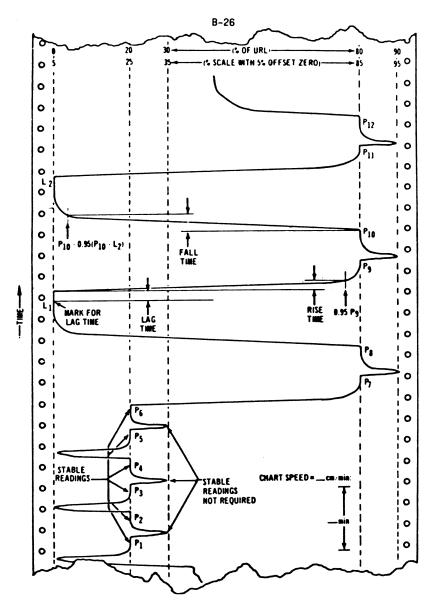


Figure B-1. Example showing the nature of the tracing obtained during the test for drift, lag time, rise time, fall time, and precision. The time scale has been greatly compressed.

(9) Test procedure. (i) Arrange to generate pollutant test atmospheres as follows:

Test atmosphere	Pollutant concentration (percent)
A ₀	Zero air. 20±5 of the upper range limit. 30±5 of the upper range limit

Test atmosphere	Pollutant concentration (percent)
A ₈₀	80±5 of the upper range limit. 90±5 of the upper range limit.

Test atmospheres A_0 , A_{20} , and A_{80} shall be consistent during the tests and from day to day.

- (ii) For steps (xxv) through (xxxi) of this section, a chart speed of at least 10 centimeters per hour shall be used. The actual chart speed, chart speed changes, and time checks shall be clearly marked on the chart.
- (iii) Allow sufficient time for test analyzer to warm up and stabilize at a line voltage of 115 volts and a room temperature of 25 °C. Recalibrate, if necessary, and adjust the zero baseline to 5 percent of chart. No further adjustments shall be made to the analyzer until the end of the tests on the third day.
- (iv) Measure test atmosphere A_0 until a stable reading is obtained, and record this reading (in ppm) as Z'_n , where n=0 (see Figure B-4 in appendix A).
- (v) Measure test atmosphere A_{20} . Allow for a stable reading and record it as M'_n , where n = 0.
- (vi) Measure test atmosphere A_{80} . Allow for a stable reading and record it as S'_{n} , where n = 0.
- (vii) The above readings for Z'_0 , M'_0 , and S'_0 should be taken at least four (4) hours prior to the beginning of test day
- (viii) At the beginning of each test day, adjust the line voltage and room temperature to the values given in table B-4.
- (ix) Measure test atmosphere A_0 continuously for at least twelve (12) continuous hours during each test day.
- (x) After the 12-hour zero drift test (step ix), sample test atmosphere A_0 . A stable reading is not required.
- (xi) Measure test atmosphere A_{20} and record the stable reading (in ppm) as P_1 . (See Figure B-4 in appendix A.)
- (xii) Sample test atmosphere A_{30} ; a stable reading is not required.
- (xiii) Measure test atmosphere A_{20} and record the stable reading as P_2 .
- (xiv) Sample test atmosphere A_0 ; a stable reading is not required.
- (xv) Measure test atmosphere A_{20} and record the stable reading as P_3 .

- (xvi) Sample test atmosphere A_{30} ; a stable reading is not required.
- (xvii) Measure test atmosphere A_{20} and record the stable reading as P_4 .
- (xviii) Sample test atmosphere A_0 ; a stable reading is not required.
- (xix) Measure test atmosphere A_{20} and record the stable reading as P_5 .
- (xx) Sample test atmosphere A_{30} ; a stable reading is not required.
- (xxi) Measure test atmosphere A_{20} and record the stable reading as P_6 .
- (xxii) Measure test atmosphere A_{30} and record the stable reading as P_7 .
- (xxiii) Sample test atmosphere A_{90} ; a stable reading is not required.
- (xxiv) Measure test atmosphere A_{80} and record the stable reading as P_8 . Increase chart speed to at least 10 centimeters per hour.
- (xxv) Measure test atmosphere A_0 . Record the stable reading as L_1 .
- (xxvi) Quickly switch the test analyzer to measure test atmosphere A_{80} and mark the recorder chart to show the exact time when the switch occurred.
- (xxvii) Measure test atmosphere A_{90} and record the stable reading as P_{80} .
- (xxviii) Sample test atmosphere A_{90} ; a stable reading is not required.
- (xxix) Measure test atmosphere A_{80} and record the stable reading as P_{10} .
- (xxx) Measure test atmosphere A_0 and record the stable reading as L_2 .
- (xxxi) Measure test atmosphere A_{80} and record the stable reading as P_{11} .
- (xxxii) Sample test atmosphere A_{90} ; a stable reading is not required.
- (xxxiii) Measure test atmosphere A_{80} and record the stable reading as P_{12} .
- (xxxiv) Repeat steps (viii) through (xxxiii) of this section, each test day.
- (xxxv) If zero and span adjustments are made after the readings are taken on test days 3, 6, 9, or 12, complete all adjustments; then measure test atmospheres A_0 , A_{80} , and A_{20} . Allow for a stable reading on each, and record the readings as $Z'_nS'_n$, and M_n respectively, where n = the test day number.
- (10) Determine the results of each day's tests as follows. Mark the recorder chart to show readings and determinations.
- (i) Zero drift. (A) 12-hour. Examine the strip chart pertaining to the 12-

hour continuous zero air test. Determine the minimum (Cmin.) and maximum (Cmax.) readings (in p/m) during this period of 12 consecutive hours, extrapolating the calibration curve to negative concentration units if necessary. Determine the 12-hour zero drift (12ZD) as $12ZD = C^{\max} - C^{\min}$. (See Figure B–5 in appendix A.)

- (B) Calculate the 24-hour zero drift (24ZD) for the n-th test day as $24ZD_n = Z_n Z_{n-1}$, or $24ZD_n = Z_n Z'_{n-1}$ if zero adjustment was made on the previous day, where $Z_n = \frac{1}{2}(L_1 + L_2)$ for L_1 and L_2 taken on the n-th test day.
- (C) Compare 12ZD and 24ZD to the zero drift specification in table B–1. Both 12ZD and 24ZD must be equal to or less than the specified value to pass the test for zero drift.
- (ii) $Span \ drift$. (A) Span drift at 20 percent of URL (MSD)

$$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$$

$$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$M_n = \frac{1}{6} \sum_{i=1}^{6} P_i$$

n indicates the n-th test day, and i indicates the i-th reading on the n-th day.

(B) Span drift at 80 percent of URL (USD):

$$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$$

or

$$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$$

n indicates the n-th test day, and i indicates the i-th reading on the n-th test day.

- (C) Both USD and MSD must be equal to or less than the respective specifications given in table B-1 to pass the test for span draft.
- (iii) Lag time. Determine, from the strip chart, the elapsed time in minutes between the mark made in step (xxvi) and the first observable (two times the noise level) response. This time must be equal to or less than the time specified in table B-1 to pass the test for lag time.
- (iv) Rise time. Calculate 95 percent of reading P_9 and determine from the recorder chart, the elapsed time between the first observable (two times noise level) response and a response equal to 95 percent of the P_9 reading. This time must be equal to or less than the rise time specified in table B–1 to pass the test for rise time.
- (v) Fall time. Calculate five percent of $(P_{10}-L_2)$ and determine, from the strip chart, the elapsed time in minutes between the first observable decrease in response following reading P_{10} and a response equal to five percent of $(P_{10}-L_2)$. This time must be equal to or less than the fall time specification in table B-1 to pass the test for fall time.
- (vi) Precision. Calculate precision (P_{20} and P_{80}) for each day's test as follows:

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^{6} P_i^2 - \frac{1}{6} \left(\sum_{i=1}^{6} P_i \right)^2 \right]}$$

(B)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$$

(C) Both P_{20} and P_{80} must be equal to or less than the specification given in table B-1 to pass the test for precision.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52694, Dec. 1, 1976; 75 FR 35600, June 22, 2010]

Pt. 53, Subpt. B, App. A

APPENDIX A TO SUBPART B OF PART 53— OPTIONAL FORMS FOR REPORTING TEST RESULTS

TABLE B-5—SYMBOLS AND ABBREVIATIONS

B _L	Analyzer reading at specified LDL concentra-
	tion.
B _z	Analyzer reading at 0 concentration for LDL test.
DM	Digital meter.
C _{max}	Maximum analyzer reading during 12ZD test.
C _{min}	Minimum analyzer reading during 12ZD test.
i	Subscript indicating the i-th quantity in a series.
IE	Interference equivalent.
L ₁	First analyzer zero reading for 24ZD test.
L ₂	Second analyzer zero reading for 24ZD test.
M _n	Average of $P_1 \dots P_6$ for the <i>n</i> -th test day.
M'_n	Adjusted span reading at 20 percent of URL on
	the n-th test day.
MSD	Span drift at 20 percent of URL.
n	Subscript indicating the test day number.
P	Analyzer reading for precision test.
P _i	The <i>i</i> -th analyzer reading for precision test.

TABLE B-5—SYMBOLS AND ABBREVIATIONS—Continued

ion at 20 percent of <i>URL</i> . on at 80 percent of <i>URL</i> . ter reading of pollutant alone for <i>IE</i> test. ter reading with interferent added for <i>IE</i>	P ₂₀ P ₈₀ R R _I
h <i>DM</i> reading for noise test.	r _i S
value (S) measured at 0 concentration. value (S) measured at 80 percent of	S ₀ S ₈₀
ge of $P_7 \dots P_{12}$ for the <i>n</i> -th test day. ed span reading at 80 percent of <i>URL</i> on	S_n S_n
range limit. drift at 80 percent o ge of L_1 and L_2 . ge of L_1 and L_2 on the n -th test day. ed zero reading on the n -th test day. rift. If zero drift. If zero drift.	URL USD Z Z _n ZD 12ZD 24ZD
h DM reading for noise test. and deviation of noise readings. value (S) measured at 0 concentration. value (S) measured at 80 percent or value (S) measured at 80 percent or value (S) measured at 80 percent of URL or L o	Fraction Fraction

Applicant	Date
Test No	
Analyzer	Range

BEADING		0% of	URL	80% of	URL
READING NUMBER (i)	TIME	DM Reading	r _į , ppm	DM Reading	r _i , ppm
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					·
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
$\sum_{i=1}^{25} r_i$					
$\sum_{i=1}^{25} r_i$ $\sum_{i=1}^{25} r_i^2$					
s			S ₀ =		S ₈₀ =

Figure B-2. Form for noise data.

																	1
Juga Area											- Range -						1
-	TEST	READING OR							Ē	TEST NUMBER							
S E	E E	CALCULATION	-	7	•	•	s	•	^	-	-	=	Ξ	21	=	=	=
<u> </u>	WE &	B ₂															
73.T	DETECTABLE	В													Γ		T
<u>'</u>		LDL:B _L ·B ₂															T
		R,				T	1	T	Γ	T		T	Ī				I
	-	R. I.							Γ								T
		IE, = R, 1 - R,															
		æ					-										
	~	78						Г									
		IE2 : R ₁₂ · R ₂															
		Ę.															Π
NCE	_	.															
FERE SJAV		IE3 * R ₁₃ · R ₃															
N3TM IUQ3		R															
4	•	R.															
		IE4 * R14 · R4															
		R															
		. R. S.															
		IEs * R _{IS} · R _S															
·	TOTAL	· (2, 73)															
											•						

Figure B-3. Form for data and calculations for lower detectable limit and interference equivalent.

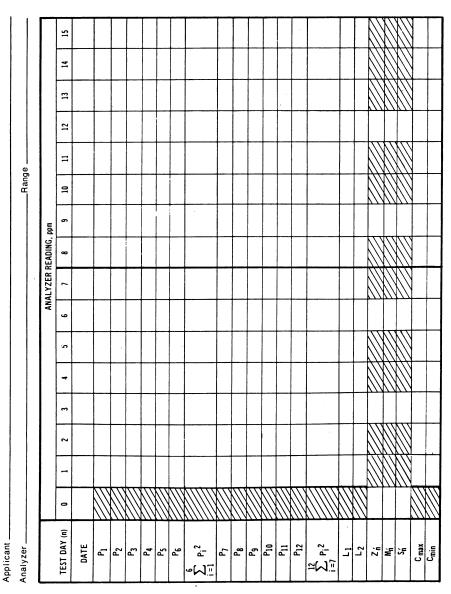
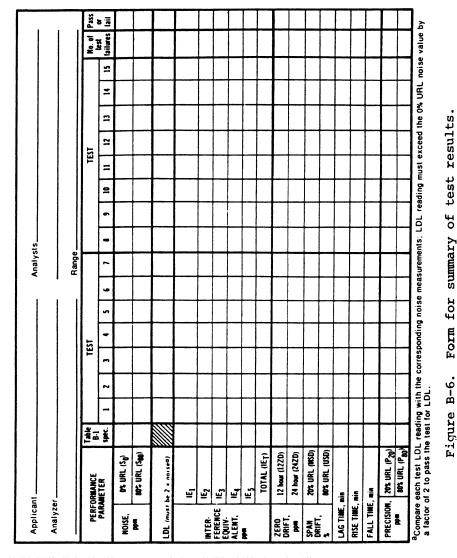


Figure B-4. Form recording data for drift and precision.

Figure B-5. Form for calculating zero drift, span drift and precision.

7 13 15 2 n - th TEST DAY $P_{80} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i 2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$ $MSD_n = \frac{M_n - M_n - 1}{M_n - 1} \times 100\%$ $MSD_n = \frac{M_n - M'_n - 1}{M'_n - 1} \times 100\%$ $USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$ $USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$ CALCULATION $12\mathrm{ZD} = \mathrm{C}_{\mathrm{max}} - \mathrm{C}_{\mathrm{min}}$ $24ZD_n = Z_n - Z_{n-1}$ $24\mathrm{ZD_n} = \mathrm{Z_n} - \mathrm{Z_{n-1}'}$ $M_n = \frac{1}{6} \sum_{i=1}^6 P_i$ $Z = \frac{1}{2}(L_1 + L_2)$ P 20=1 TEST PARAMETER 20% URL 80% URL 20% URL 24 hour 80% URL 12 hour Analyzer Applicant Span drift Precision Zero drift



 $[40~{\rm FR}~7049,~{\rm Feb}.~18,~1975,~{\rm as~amended~at}~40~{\rm FR}~18169,~{\rm Apr.}~25,~1975]$

Subpart C—Procedures for Determining Comparability Between Candidate Methods and Reference Methods

Source: 71 FR 61278, Oct. 17, 2006, unless otherwise noted.

§53.30 General provisions.

(a) Determination of comparability. The test procedures prescribed in this subpart shall be used to determine if a candidate method is comparable to a reference method when both methods measure pollutant concentrations in